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ARTICLE XXIV.

Chemical Examination of two Minerals from the neighbourhood of Reading, Pa.; and on the occurrence of Gold in Pennsylvania. By Charles M. Wetherill, Ph. D.

1. *Molybdenite*.—This mineral, specimens of which were given to me by Dr. Bischoff and Geo. M. Keim, Esq., of Reading, is found in abundance at the Zion Church, Alsace, in the neighbourhood of that city. It occurs of considerable purity in plates and scales in a quartz matrix. The colour of the latter is like plumbago, but more brilliant. The streak on paper, that of plumbago; on porcelain, olive green. It is impressible to the nail, giving a hardness of 1 by Mohs' scale. 0.7255 grammes weighed in water, 0.565, corresponding to a density of 4.52. The following is its behaviour before the blowpipe. Heated alone on charcoal, white fumes were evolved which coated the coal, and the specimen was left tarnished. Heated alone in the open tube, sulphurous acid was given off. In the platinum forceps, it coloured the outer flame yellowish green. On the platinum wire in the outer flame, gave a glass, yellowish when hot, colourless on cooling; in the inner flame, the glass was greenish. Did not readily dissolve in this bead of microscopic salt. Borax gave similar reactions. Heated with nitre in the platinum spoon, deflagrated with formation of sulphate and molybdate of potassa. Some of the crystalline scales heated in the closed tube gave off a small quantity of water, the quantitative determination of which was effected by raising to a bright red heat in a covered crucible 0.673 grammes of the crushed mineral. The loss after ignition was 0.002, corresponding to a per centage of 0.297 water. A qualitative examination in the moist way detected as ingredients of the mineral, silica, sulphur, molybdenum and iron, the quantitative determination of which is afforded by the following analytical data. 0.2775 grammes of the mineral (which was pulverized with great difficulty, on account of its soft and lamellar nature,) after several days' digestion in aqua regia, left a white residue of silica weighing 0.008, corresponding to a per centage of 2.283. The sulphuric acid yielded on the addition of nitrate of baryta 0.7697, sulphate, which corresponds to 0.106, sulphur, or 38.198 per cent. After removing the excess of baryta salt, the iron precipitated by ammonia weighed 0.0097 or 3.495 per cent. It was proposed to determine the molybdenum as Mo O_2 by igniting the evaporated residue according to Rose, in an atmosphere of hydrogen, but an

accident towards the end of the experiment, obliged its determination by loss, which gave a percentage of 55·727. The analysis of the mineral, therefore, gives the following percentage:—

Aq	.	.	0·297
S	.	.	38·198
Si O ₃	.	.	2·283
Fe ₂ O ₃	.	.	3·495
Mo	.	.	55·727
			<hr/>
			100·000

A calculation of the per centage relations of the molybdenum and sulphur, without reference to the other ingredients gives,

S ₂	.	.	40·668
Mo	.	.	59·332
			<hr/>
			100,000

Dana gives the per centage calculated from the formula MoS₂=sulphur 40·99, and molybdenum 59·01, and Weber in his tables to Rose's analysis, (late edition,) gives for the same calculation, sulphur 41·123, and molybdena 58·877.

2. *Zircon*.—Last summer, Mr. Isaac Lea exhibited to the Academy of Natural Sciences, specimens of Zircon imbedded in magnetic iron, which he obtained from a heap of ore at Eckards' iron furnace, and which was said to have come from a locality eight or nine miles from Reading, N. E. of Pricetown. I received specimens from Mr. Lea, and also from Dr. Heister, from the same heap. Mr. Geo. M. Keim visited, at my request, the locality from whence the ore was obtained, and sent me specimens, and also a few of the same kind which he found in the Mineral Spring Valley, just outside of Reading. The Zircon occurs in large crystals firmly imbedded in the ore, which are in some instances well terminated, but brittle, and detached with great difficulty from their matrix, to which they adhere with such tenacity, that the impression left in the matrix after detaching them is polished, of vitreous lustre and of the colour of the crystals, as if they had been melted in the ore after their formation. Their planes and angles (as noticed also by Mr. Lea) are rounded off in places, as if they had been subjected to an incipient fusion. The largest crystals which I obtained after carefully breaking several pounds of the ore, measured one and a half inches, by one-quarter inch, by three-eighth inch (nearly.) It was distinctly terminated at one end, and showed traces of termination at the other. The usual crystal form was a right prism terminated by corresponding pyramids, the angles of which were frequently modified. The colour, chocolate brown; opaque; lustre, adamantine; planes, as before stated, uneven. One specimen which was too much broken to form certain conclusions, appeared to be part of one of the terminal pyramids. It was highly modified, possessed perfectly sharp edges and glass smooth planes, of adamantine lustre; was on the

edges transparent, like hyacinth, and of deeper colour than the other crystals. The cleavage of the mineral was indistinct and fracture very uneven, but apparently in planes perpendicular to the vertical axis. Hardness, between quartz and topaz, or 7—8 of Mohs' scale. Its powder was brownish yellow, very light. 0.965 grammes weighed in distilled water of temperature 26° C., 0.755 corresponding to a density of 4.595. Before the blowpipe alone in the forceps, and on coal, infusible, *deepens* in colour. The crystals heated to redness in a closed tube give off water, and are unchanged in colour. Does not tinge the blowpipe flame. With borax and salt of phosphorus, gives the reaction of iron. No reaction for manganese on platinum foil with carbonate of soda and nitre, but yields an opaque reddish white mass. Is not attacked by acids. A portion of the mineral finely powdered by washing over, tested for potassa and soda after attacking by concentrated hydrofluoric acid, gave negative results. An examination by the moist way, detected silica, zirconia, iron. The iron present tinged the zirconia green when precipitated by ammonia and hydrosulphuret of ammonium. In a preliminary experiment 0.8985 grammes of the finely powdered mineral exposed to a white heat in a platinum crucible, lost 0.005 which corresponds to a per-centage of 0.556 water. It will be perceived that in its behaviour, this mineral corresponds, with one exception, to zircon, or anhydrous silicate of zirconia. Its water would assimilate it to malakon $2(\text{Zr}_2\text{O}_3\text{SiO}_3) + \text{HO}$, but it contains less water. Malakon has a lower hardness and sp. gr. than zircon, but by ignition its density is raised to that of zircon. It is supposed by some mineralogists, that malakon is zircon slightly altered by the absorption of water, and by Damour, that water constitutes the difference between them, with which malakon is chemically combined. An analysis of the present mineral is, therefore, interesting, as perhaps throwing light upon the nature of these two species.

The analysis of zircon presents well known difficulties in the separation of its silica and zirconia. According to Scheerer, when water is added to the result of fusing the mineral with carbonate of soda, a gritty powder remains, which is a combination of soda and zirconia, and which by treatment with concentrated hydrochloric acid becomes gelatinous. Gibbs has, however, shown, that besides the above mentioned constituents, this compound contains 24.76 silica, and, that if, after attacking by hydrochloric acid to decompose it, the solution be evaporated to dryness for determining the silica in the usual manner, this silica will be found to contain 30.15 per cent. of zirconia. In an analysis where the silica was determined in the usual manner, I found it contained, beside a small quantity of zirconia, other impurities. In this analysis one gramme of the elutriated mineral (dried at a steam heat) was ignited in a platinum crucible, and the water obtained by loss. Four times the weight of the mineral of carbonate of soda, with the addition according to Henneberg's suggestion of one-eighth nitre, were mingled with the residue in the crucible and exposed to a white heat. The result was found to be well melted, and was treated by water and hydrochloric acid. If sufficient water be present, the acid acts in the cold upon the granular silicate of zirconia soda, perceptibly to the eye, leaving white flakes of silica without the least grittiness. The hydrochloric solution was evaporated with the water-bath to complete dryness, and the residue treated with hydrochloric acid and water.

Since the residue appeared to contain impurities in the silica, dilute sulphuric acid was added, and the whole evaporated until acid fumes began to be evolved. After cooling, water was added, and the silica separated by filtration. To the filtrate was added ammonia and hydrosulphuret of ammonium, the zirconia and sulphuret of iron, after settling, removed by decanting and the iron separated from the zirconia by sulphurous acid according to Berthier's method. The zirconia was, after boiling, filtered and washed, and after decomposing the sulphites and hydrosulphites by nitric acid, the iron was separated from the filtered liquid by ammonia. The addition of phosphate of soda to the filtrate from the oxide of iron, and suffering the beaker to stand in a warm place for several days, gave negative results as to the presence of magnesia. The silica in this analysis was treated twice by concentrated hydrofluoric acid, (which was pure, leaving no residue when evaporated,) and a residue was obtained which, after heating with sulphuric acid weighed 0.038. On boiling this with dilute sulphuric acid to the evaporation of part of the acid, 0.0095 of zirconia was obtained, and 0.0075 silica, which together equal 0.017, leaving unaccounted for 0.021, possibly soda or its salt, in combination with the silica and zirconia. Subtracting the zirconia from the silica, and adding it to that already obtained, this first analysis on one gramme, gave the following per centage results:—

Silica,	.	.	.	36.72
Zirconia,	.	.	.	65.45
Peroxide of iron,	.	.	.	2.07
Water,	.	.	.	0.50
				<hr/>
				104.74

Both silica and zirconia are here evidently too high. The former two per cent. from the probable presence of alkali; the zirconia also two per cent., from a cause to be mentioned later.

The difficulties presenting themselves in the analysis of zircon are owing, as is well known, to the facility with which its zirconia forms insoluble compounds with silica, and the re-agents necessary to effect its separation. From my experiments on this mineral it would appear, that even the low temperature of the steam bath when water is absent, has a tendency to the formation of basic zirconia compounds, not *readily* soluble even in sulphuric acid. On the supposition that this would not take place by presence of sulphuric acid in considerable excess before evaporating to dryness, and that its silica would be, perhaps, rendered insoluble by the same temperature, I modified its analysis in the following manner, which would appear to be a speedy and accurate method for the analysis of zircon. One gramme of the elutriated mineral, after determining the water by ignition, was fused with four times its weight of carbonate of soda without addition of nitre. Ten minutes' exposure to the heat obtained by forcing a current of air by the table blowpipe through a gas argand flame was sufficient to effect a complete decomposition. To the residue was added a considerable quantity of water, then hydrochloric acid in the cold, without first separating the granular zirconia soda as proposed by Scheerer. The silica remains as pure white flocculæ without any grit. An excess of sulphuric acid is now added and the whole evaporated until fumes of the acid are evolved, at which temperature it is kept for some time. It would be, perhaps, better to add sulphuric acid at

once to the result of the fusion with carbonate of soda and addition of water, and omit the use of hydrochloric acid altogether. The silica filtered rapidly, and after the incineration was almost completely dissolved in hydrofluoric acid. The zirconia and iron were determined as in the first analysis. The zirconia requires a long washing, but filters quickly. Care must be taken in the incineration of this zirconia, otherwise a too high result will be obtained as in the first analysis. When the precipitate is burned in the usual manner, at a low red heat, and until the filter ash is perfectly white, the zirconia remains in shining lemon-coloured irregular masses, which when exposed to a high temperature lose weight. Thus, in my second analysis with one gramme of substance, the weight of the zirconia ignited in the ordinary way, was 0.641, and by successive ignitions at a white heat, was 0.636—0.635—0.635. When it had no longer lost weight, it had a beautiful pearl gray lustre, scratched glass, and gave a brilliant light when heated before the blowpipe. The analysis by this method upon one gramme of the mineral, gave the following per centage results:—

Silica,	.	.	.	34.07
Zirconia,	.	.	.	63.50
Peroxide of iron,	.	.	.	2.02
Water,	.	.	.	50
				<hr/>
				100.09

The whole crystals of zircon were also raised to a white heat in a platinum crucible, until no further loss of weight—0.997 lost 0.0035, corresponding to a per centage of 0.35 water being less than that obtained from the finely pulverized mineral. The heat was raised gradually, but no glowing of the crystals was observed. After this ignition, they were in lustre more brilliantly adamantine, and darker; although in spots they appeared lighter than originally, as if the iron were not uniformly diffused throughout. As the mineral occurs in a matrix of iron ore, and has apparently been subjected to a high temperature therein, it would seem as if a large part of this iron had been absorbed by cementation. It follows from the analysis of this mineral, that the contents of water alone will not serve as a distinguishing characteristic between malakon and zircon, as Damour proposes; nor that malakon is zircon, altered by the absorption of water as Dana deems possible; but, that the two species are distinct, and differ probably in containing zirconia in two allotropic conditions, as believed by Scheerer. The following are analyses of the two minerals. 1st, Zircon by Henneberg. 2d, Zircon from Litchfield, by Gibbs. 3d, Malakon from Hitteröe, by Scheerer. 4th, Malakon from Chanteloube, by Damour.

	1	2	3	4
Silica,	33.85	35.26	31.31	31.23
Zirconia	64.81	63.33	63.40	61.70
Iron Perox	1.55	0.79	0.41	2.91
Water	—	—	3.03	3.29
Ytria	—	—	0.34	—
Lime	0.88	—	0.39	trace
Magnesia	—	—	0.11	—
Undecomposed . . .	—	0.39	—	trace manganese.
				<hr/>
				99.13
				<hr/>
				99.74
				<hr/>
				98.99
				<hr/>
				101.09

W. Henneberg found in his experiments on the effect of heat on zircon, that the condensation after ignition, was as 10,000 to 9798. The density before being 4.615, and after 4.71; and also observed, that at a certain stage, the crystals exhibited a phosphorescence. In my experiments, raising the crystals to a white heat in a platinum crucible, I did not remark any phosphorescence, which was carefully looked for, and the temperature gradually raised. The density of the crystals before and after ignition, was 4.595 and 4.62, corresponding to a condensation as 10,000 to 9946.

Rosé, in the last edition of his *Handbuch*, suggests the fusion with bisulphate of ammonia, which has never been applied to zircon, as a probable means of its analysis. I performed the experiment very carefully upon 0.961 grammes of the finely pulverized mineral, keeping it as long as possible in contact with the re-agent; but with negative results. The insoluble portion was silicate of zirconia, very white and almost entirely free from iron, while about 1.5 per cent. of zirconia dissolved out, and was precipitated along with the iron. This method, though not applicable to the analysis, may serve to free silicate of zirconia from iron, should it be required to prepare this salt from impure zircon.

3. *On the Occurrence of Gold in Pennsylvania.*—In the spring of 1851, an earth was given to me for examination, of which the locality was not exactly stated, but which was said to have been taken not far from the city, in which gold was detected. The earth was said to have been obtained in digging a well. Several months later, while in Reading, I met with a notice in a German newspaper of that place, which stated that some time previously an earth had been found in digging a well, upon the land of Mr. Yoder, Franconia township, Montgomery county, which proved, upon examination, to contain gold. I have no doubt but that this is the locality of the earth which I examined. Several rocks from the neighbourhood were submitted, consisting of clay slate rock, ferruginous quartz, decayed in places, containing pyrites and magnetic oxide of iron sand. In most of these gold was detected in traces. Some specimens contained no gold whatever. The earth from the well, which was more particularly examined, consisted of sand and gravel, coating in some places fragments of shale or other rock. A careful examination of these with the lens, detected a rather thick spangle of gold adhering to the gravel, and a small rounded mass of a white malleable metal which proved by a microchemical investigation upon half of it to be native tin, which occurs only, according to Dana, in small grayish white metallic grains along with Siberian gold. It melted before the blowpipe, was oxidized by nitric acid, the resulting oxide being insoluble in tartaric acid. Dissolved slowly in HCl, to which solution H₂S gave the yellowish brown precipitate SnS+SnS₂. This occurrence of native tin is strongly opposed to the supposition of fraud in the earth examined. Separating the rock and washing, gave a further quantity of gold spangles very fine, and mingled with pyrites and magnetic oxide of iron, together with more spangles of native tin. One and a half pounds of the original substance, from which these spangles were removed, after separation of the rocks and concentration by washing, were melted with twice their weight of litharge, (previously tested for gold,) and a small quantity of charcoal powder. The resulting button of lead was cupelled (adding to the lead the gold already found) and the

silver treated with nitric acid which left a coherent mass of gold weighing 0·006 grammes. One hundred pounds of the earth would, therefore, contain 0·4 grammes of gold, worth about twenty-six and a half cents.

During a stay at Reading, in the summer of 1851, I noticed a vein of decayed ferruginous quartz, very much resembling the auriferous quartz of North Carolina. It was uncovered in exploring the deposits of iron ore in Penn's Mount behind the city. I neglected at the time to secure specimens, and upon a second visit to the locality this spring, to obtain a quantity for analysis, I found it covered. I obtained, however, from the vicinity a quartz rock, quartz and felspar mingled, and sand, which, on analysis, yielded an exceedingly minute quantity of a brownish powder after treating the silver button resulting from cupellation by nitric acid; but which were too minute from which to derive any definite conclusion as to the presence or absence of gold. A former pupil of mine in an examination of the pyrites of the same locality, thought to have detected traces of gold. I have no doubt, that a more careful examination of the rocks in the vicinity would yield affirmative results in an examination for this metal.